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QUANTITY AND QUALITY OF CREOSOTE FOUND IN TWO TREATED PILES AFTER LONG SERVICE.

BY

E. BATEMAN,
Chemist in Forest Products.

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QUANTITY AND QUALITY OF CREOSOTE FOUND IN TWO TREATED PILES AFTER LONG SERVICE.

INTRODUCTION.

This circular gives the results of analyses of the quantity and quality of creosote found in two treated piles[1] after long service. The piles were of pine (probably longleaf), and had been in the teredo-infested waters of the Gulf of Mexico for about 30 years. One was perfectly sound, but the other had been attacked, particularly at the water line. The analyses were made to determine whether the difference in their durability could be accounted for by a difference in the amount or character of creosote in the wood.

The piles had been treated by the old Bethell process, but no treating records are available, nor is it known where the creosote was obtained.

DESCRIPTION OF SPECIMENS.

Pile No. 1.—This pile, said to have been in service 30 years, was perfectly preserved, showing no indications of decay nor of attack by teredo. The portion above water was badly checked. It was received in three sections: Section 1, taken from above the water line; section 2, taken from near the mud line; section 3, taken from the lower end of the pile in the mud.

Pile No. 2.—This pile, which had been creosoted and placed in the Biloxi Bay trestle in 1879, and removed in July, 1910, had been attacked by teredo, especially near the water line. Only a portion of the whole pole, approximately 6 feet long, extending 3 feet above and 3 feet below the water line, was received. This will be considered as three sections: Section 1, above the water line; section 2, at the water line; section 3, below the water line.

Plate I, figure 1, shows radial views of the three sections of pile No. 1. It brings out clearly the depth of penetration of the creosote. Plate I, figure 2, shows three cross sections of pile No. 2. The work of the teredo, it will be noted, is in the treated wood.

[1] Shipped to Forest Products Laboratory by Mr. J. B. Lindsey, superintendent, West Pascagoula Creosoting Works, Gautier, Miss., in July, 1910.

EXAMINATION FOR THE QUANTITY OF CREOSOTE PRESENT.

Samples from the several portions of the piles were taken by borings distributed over the entire cross section. A weighed portion of the average samples from each section thus obtained was treated with chloroform; and the loss in weight after treatment and drying was determined. The loss includes all the creosote, all the rosin, and all the moisture. The extracted material was then treated with a sodium carbonate solution, and the dissolved rosin was recovered by precipitating it with acid. Moisture determinations were made on separate portions of the original sample. Then the amount of rosin and moisture was subtracted from the total of creosote, rosin, and water. The result was calculated in pounds of creosote per cubic foot of wood. Measurements of the relative proportions of the treated and untreated area of each cross section were then made. From these measurements and the previous calculations estimates were made of the quantity of creosote in the treated portion only. The results obtained are given in Table 1.

TABLE 1.—*Quantity of creosote in two piles.*

Pile No.	Section No.	Amount of creosote found per cubic foot.	
		Entire cross section.	Treated portion only.
		Pounds.	*Pounds.*
1	1	2.6	4.5
1	2	10.7	15.3
1	3	12.0	17.1
2	1	10.4	17.0
2	2	5.8	16.5
2	3	11.5	17.9

In the case of pile No. 2 the amount of creosote in the entire cross section at the water line (sec. 2) is only about half of that in either of the other two sections; but when calculated for the treated portion only it is nearly the same. This difference is due to the loss of a great portion of the creosoted wood in this section, making the proportion of untreated to treated wood much higher than in the other two cases. The proportion of the treated area in the three sections of this pile as received at the laboratory was: Section 1, 61 per cent treated; section 2, 35 per cent treated; section 3, 64 per cent treated.

ANALYSIS OF EXTRACTED OILS.

To determine the quality of the creosote, the oil was extracted from a large volume of chips by chloroform. The resulting extract was then freed from rosin by the use of sodium carbonate, and from

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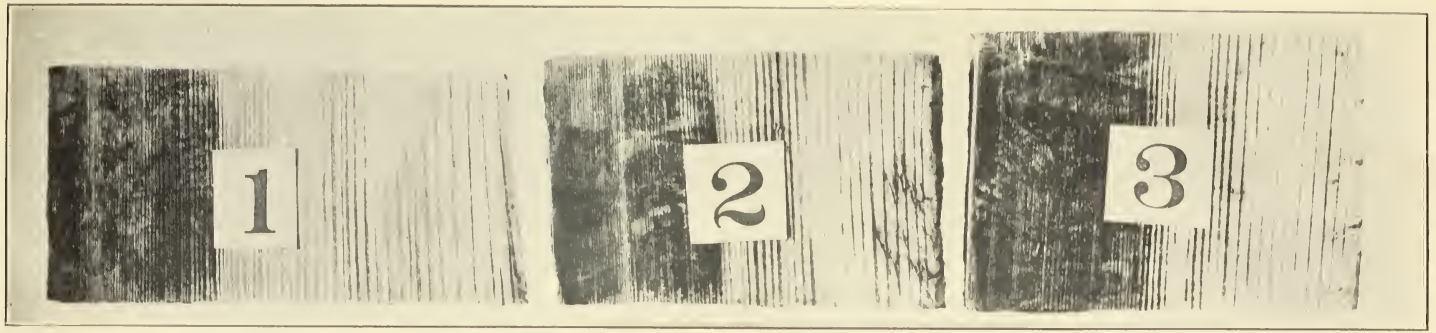

FIG. 1.—RADIAL VIEWS OF THREE SECTIONS FROM PILE NO. 1.

[1, above water line; 2, in the water; 3, in the mud.]

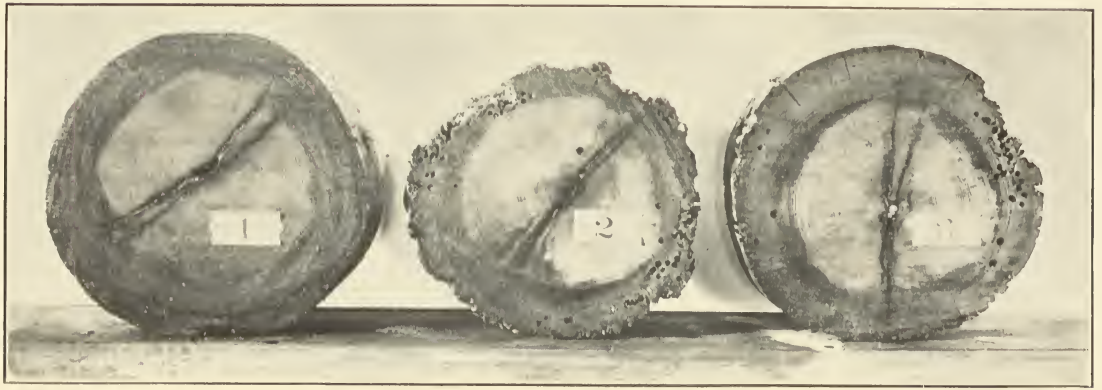

FIG. 2.—CROSS SECTIONS OF PILE NO. 2.

[1, above water line; 2, at water line; 3 below water.]

chloroform by distillation. The residual creosote was then analyzed according to the method described in Forest Service Circulars 112 and 191. The results of these analyses are given in Tables 2 and 3.

TABLE 2.—*Results of fractional distillation and index of refraction determinations of creosote extracted from pile No. 1.*

Temperature.	Percentage weight of distillate.			Average index of refraction of 2 and 3.
	Section 1.	Section 2.	Section 3.	
°C.				
205	2.0	.....	2.5	.....
215		2.7	2.0	.....
225		15.5	26.3	.....
235	8.2	12.0	8.5	1.5922
245		3.1	2.5	1.5920
255	13.7	2.1	2.0	1.5921
265		2.6	1.9	1.5939
275	6.6	2.4	2.5	1.5981
285	5.0	3.2	3.3	1.6041
295	6.2	4.1	3.5	1.6123
305	6.0	4.8	4.3	1.6203
320	10.5	8.6	7.9	1.6310
Residue.	41.6	38.4	32.3	.....

TABLE 3.—*Results of fractional distillation and index of refraction determinations of creosote extracted from pile No. 2.*

Temperature.	Percentage weight of distillate.			Average index of refraction of 2 and 3.
	Section 1.	Section 2.	Section 3.	
°C.				
235	4.0	4.6	6.0	1.5795
245	1.8		3.4	1.5825
255	1.6		3.2	1.5842
265	2.1	.....	8.9	1.5872
275	2.1			
285	3.0	4.2	9.2	1.5945
295	2.4	2.2	3.1	1.5997
305	1.0	2.6	.....	.....
Residue.	81.2	85.8	64.4	.....

None of the sections contained an appreciable amount of light oils. The creosote from section 3 of pile No. 1, in which presumably less change in the character of the oil had occurred, contained 2.5 per cent of oils distilling below 205°. The same creosote contained over 40 per cent of naphthalene oils (distilling between 205° and 255° C.). The other two sections of the same pile also contained considerable quantities of naphthalene.

The distillation of the creosote from pile No. 2 gave a very small percentage of distillate (below 305° C.) and a large amount of residue. The oil from section 3 (below the water line) of this pile, which yielded the largest amount of distillate, contained only 12.6 per cent of oils volatile below 255° C., and little or no naphthalene.

Sulphonation tests carried out on the fraction from 285° to 305° C. and 305° to 320° C. of the creosote from pile No. 1 failed to give any sulphonation residue. This oil resembles an imported creosote oil and is probably a pure coal-tar product. The index of refraction

values of the fractions above 295° C. are a little low, but this could easily be due to the presence of a small amount of rosin which had escaped separation before the oil was analyzed. The color, odor, and character of the fraction were like those of coal-tar creosote.

Sulphonation tests on the portions 285° to 305° C. of the creosote from pile No. 2 yielded a sulphonation residue of 2.6 per cent. The

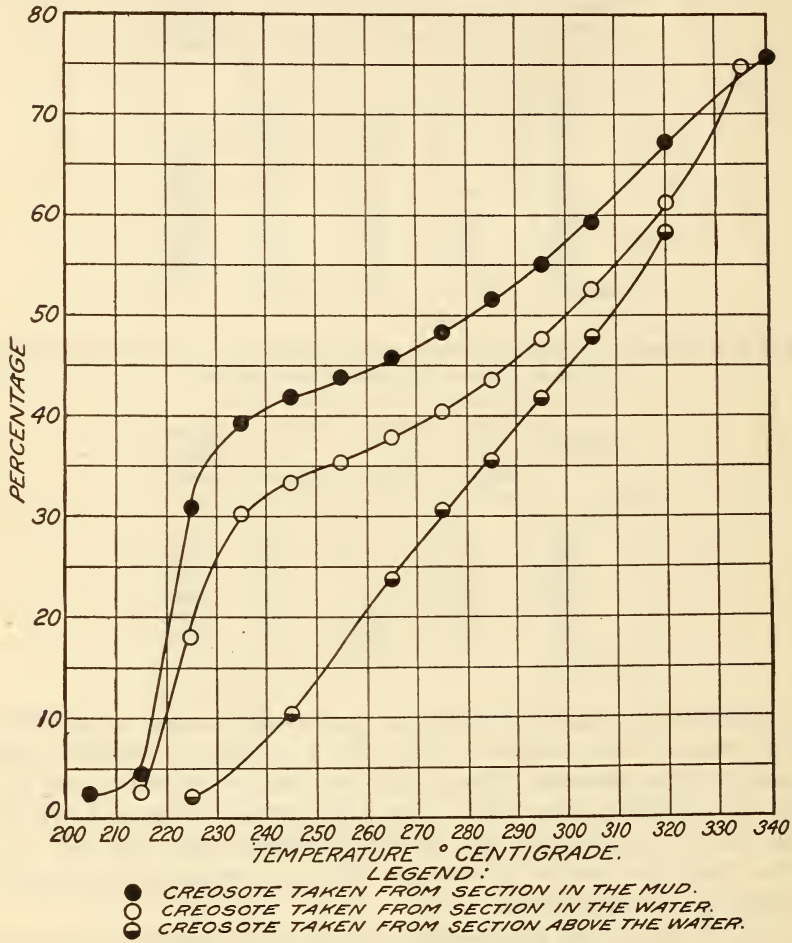

FIG. 1.—Fractional distillation curves, pile No. 1.

index of refraction values, as well as the color and odor, of this oil resemble those of water-gas-tar creosote.

CHANGE IN COMPOSITION OF THE CREOSOTE BY EXPOSURE.

Inspection of Table 2 indicates that whatever loss of creosote occurred in the several sections through leaching and volatilization while the piles were in use must have occurred in the lighter frac-

tions. If we assume that no change has occurred in the creosote extracted from section 3 of pile No. 1 (the portion buried in the mud) and that no change has occurred in the higher boiling fractions of the oil from the other two sections of this pile, the loss of oil in sections 1 and 2 may be computed as follows:

The fractions above 275° C. and residue for section 3 amounts to 51.3 per cent of the total; the same portion of the creosote extracted from section 2 amounts to 59.1 per cent. If the oil from section 3 is unchanged, as assumed above, the original volume of the oil in section 2 can readily be obtained by the proportion $59.1 : x = 51.3 : 100$, or $x = 115$ per cent. That is, the creosote extracted from section 2 is the residual of an oil that was originally 15 per cent greater in volume. By a similar computation it is found that the creosote extracted from section 1 is the residual of a creosote originally 35 per cent greater in volume.

The change in composition of the creosote from sections 1 and 2 is shown more fully in Table 4, in which the fractional distillation is computed on the basis of percentage of what is assumed to be the original oil.

TABLE 4.—*Fractional distillation of creosote extracted from pile No. 1; in percentage of assumed original oil.*

Temperature.	Percentage weight of distillate.		
	Section 3.	Section 2.	Section 1.
°C.			
225	30.8	15.8	1.5
245	11.0	13.1	6.1
275	6.4	6.2	15.0
320	19.0	18.0	20.5
Residue.	32.3	33.4	30.8

Table 4 shows that the loss of creosote in that portion of the pile in the water as compared with the loss from the portion buried in the mud was confined to the fraction distilling below 225° C. and that the loss from the portion in the air occurred only in the fractions below 245° C. The small excess of distillate between 225° and 245° C. in the creosote from section 2 over that for the same fraction from section 3 may be accounted for by the effect which the absence of some of the lower boiling constituents at the lower stages of the distillation produce upon the fractionation of the distillate. The same explanation will account for part of the relatively large excess in the distillate between 245° and 275° C. of the oil from section 3.

Allowing for the losses as computed above, sections 1 and 2 of this pile originally had 6.1 and 17.6 pounds, respectively, of creosote per cubic foot of the treated portions. Section 2 thus agrees very well with section 3 (see Table 1). But the figure for section 1 is so much

at variance with the figures for sections 2 and 3 that this section probably lost creosote in such a manner as to leave the composition of the residual oil unchanged, as by "bleeding." The original volume of oil must therefore have been more than 35 per cent greater than the present.

Similar changes occurred in pile No. 2, except that in this case section 2, which is the portion at the water line, changed most. This may be due to its position, where it was subject to the influences both of sun and water; and also to the fact that, being riddled by the teredo, more opportunity was afforded for leaching of the creosote.

SUMMARY.

Practically no light oils (oils distilling below 205° C.) were found in the piles after their long period of service. If originally present, they were lost by volatilization and leaching.

The creosote in the pile which was perfectly preserved contained originally at least 40 per cent of naphthalene fractions, a large portion of which remained in the wood. The creosote in the pile, which was less perfectly preserved, contained little or no naphthalene.

The pitchy matter, which on distillation formed the residue above 320° C. (pile No. 2), is seemingly of an inert character and little objectionable to the teredo. A heavy treatment with creosote consisting largely of this material did not entirely save the pile from attack.

Loss of oil from that portion of the pile in the water, in the case of the creosote in pile No. 1, which is a pure coal-tar creosote, apparently occurred only in the fraction distilling below 225° C.

Approved:

JAMES WILSON,
Secretary of Agriculture.

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